

*SUBCO*

the silicate concentration, expressed as SiO<sub>2</sub> in the said initial base stock being lower than 100 g/l and the electrolyte concentration in the said initial base stock being lower than 17 g/l;

(ii) adding the acidifying agent [is added] to said base stock until a pH value of the reaction mixture of at least approximately 7 is obtained;

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cont.*

(iii) adding simultaneously the acidifying agent, and if appropriate, the remaining amount of the silicate [are added simultaneously] to the reaction mixture;

(B) separating from the reaction mixture a precipitation cake which has a solids content of between 10 and 40%; and

(C) deagglomerating [the] said cake to obtain a suspension of [low viscosity] agglomerates having a median diameter D<sub>50</sub> smaller than 5  $\mu\text{m}$ , whereby a suspension of low viscosity is provided.

#### REMARKS

This amendment is responsive to the Office Action dated December 8, 1997. Entry of the foregoing and favorable reconsideration of the subject application in light of the following remarks, pursuant to and consistent with 37 CFR 1.112, are respectfully requested.

The claims have been amended to more clearly define the present invention. Specifically, claim 29 has been amended in response to the rejection under 35 U.S.C. §112, second paragraph, comments (a) and (e), wherein the Examiner correctly indicated that the word "crumbled" should be before "filter." Claims 22, 31 and 39 have been amended in

response to the rejection under 35 U.S.C. §112, second paragraph, comments (b) and (d), in order to clarify allegedly awkward language and ensure that the steps recited in claims 31 and 39 agree with the preambles. And claim 38 has been amended in response to the rejection, comment (f), where the Examiner correctly notes that "solids" should be "silica." As these amendments merely corrected minor grammatical errors in the claims, no new matter has been added.

In addition, claim 39 has been amended to clarify that "low viscosity" means that the agglomerates have a median diameter  $D_{50}$  which is less than 5  $\mu\text{m}$ . Support for this amendment may be found on page 3, lines 5-8. Thus, no new matter has been added. Claims 22-46 are pending.

Claims 22-46 have been rejected under 35 U.S.C. §112, second paragraph, as allegedly being indefinite for failing to particularly point out and distinctly claim the invention. Specifically, the Office Action rejects claim 29 on the basis of clarity, with the specific comment (a) that the word "crumbled" should be before filter. Applicants respectfully thank the Examiner for pointing this out, and have made the required amendments. Applicants also believe that these amendments resolve the clarity issue mentioned under comment (e) on page 2 of the Office Action. Accordingly, this rejection as it applies to claim 29 is believed to be rendered moot.

In comment (b) on page 1 of the Office Action, claims 31 and 39 have been rejected under 35 U.S.C. §112, second paragraph, because the steps of the claimed processes do not

appear to agree with the preambles. It is believed that the above amendments rectify any issues of clarity with regard to these claims.

Comment (b) also indicates that a further step in claims 31 and 39 of adding water "appears to be necessary." In this regard, Applicants respectfully point out that water may be added, but this is not necessary.

For instance, in one embodiment, the filter cake may be converted in a suspension by washing the cake with organic solvents, drying the cake to obtain a silica powder, and subsequently suspending the silica powder in water (p. 12, l. 1-9). However, the precipitation cake contains water molecules usually referred to as "structural water" because they occupy the spaces between the silica agglomerates (i.e., see WO '330, page 4, l. 12-17). Thus a mechanical crumbling or a combination of a mechanical and chemical crumbling as disclosed in the specification (page 3, l. 18 and p. 11, l. 2) will release the structural water contained in the cake and provide a suspension. In this case, the water trapped inside the cake becomes the suspension medium.

In comment (c) on page 2 of the Office Action, claim 39 is also rejected because it is allegedly unclear as to determine whether it is "appropriate" to add the remaining amount of the silicate. In this regard, Applicants respectfully point out that claim 39 as amended includes step A(i), wherein "at least a proportion of the total amount of silicate" is added to the reaction. Applicants believe it is clear in view of this sequence of steps and in the language of the claim that the remainder of the silicate should be added during step A(iii), "if appropriate," or in other words, if only a proportion was used in step A(i). Therefor,

Applicants respectfully request that this rejection as it pertains to this limitation of claim 39 be reconsidered and withdrawn.

In comment (d) on page 2 of the Office Action, claims 22, 31 and 39 are rejected under 35 U.S.C. §112, second paragraph, for awkward wording. It is believed that the above amendments render this particular rejection moot.

Finally, in comment (f) on page 2, the Office Action suggests that perhaps "solids content" was meant to be "silica content." Claim 38 has been amended accordingly, and this rejection is believed to be rendered moot.

In view of the above amendments, reconsideration and withdrawal of all rejections under 35 U.S.C. §112, second paragraph is respectfully requested.

Claims 22-29 have been rejected under 35 U.S.C. §102(b) as allegedly anticipated by, or in the alternative, under 35 U.S.C. §103 as allegedly obvious over WO '330. Specifically, the Office Action states at page 3 that the reference teaches a silica slurry of 25% silica, and has shifted the burden to Applicants to show that the silica slurry is different from the suspension claimed in claims 22-29. Applicants respectfully traverse the rejection.

WO 90/0330 generally discloses an aqueous suspension of precipitated silica with a high solids content (40 to 60% of solids by weight) and a relatively low viscosity. The suspension may be obtained by milling a coarse suspension so as to decrease the particle size of the silica agglomerates to 0.3 to 3  $\mu\text{m}$ . However, the viscosities that are mentioned in WO '330 are in the range of 50 to 150 cps (Brookfield viscosity), i.e.,  $5 \times 10^{-2}$  to  $15 \times 10^{-2}$  Pa.s being the lowest values, even at the end of the process where diluted suspensions having

solids contents which overlap that of the instantly claimed suspension (40 cp. at a shear rate of 50 s<sup>-1</sup>) are implemented. This is in contrast to the suspensions of the present invention, which have viscosities lower than 4 X 10<sup>-2</sup> Pa.s (claim 22) or 2 X 10<sup>-2</sup> Pa.s (claim 24).

Furthermore, Applicants stress that in process steps wherein diluted silica suspensions are implemented, i.e., silica suspensions with a solid content of less than 40% as in the instant invention, the median diameter of the agglomerates is substantially greater than the median diameter of the agglomerates of the instant invention ( $D_{50} \leq 5 \mu\text{m}$ ). Indeed, the 25% silica slurry of WO '330 to which the Office Action refers has a median particle size of 19  $\mu\text{m}$  (see page 12, line 17).

Thus, the  $D_{50}$  values provided in WO '330 for diluted suspension are  $D_{50} = 7$  to  $25 \mu\text{m}$  for a solids content of 35-45% (page 7, lines 15-18), as compared with the agglomerates of the present invention, which have a median diameter  $D_{50}$  which is smaller than 5  $\mu\text{m}$  (see claim 28). It is only in the ultimate step in the high speed fluid shear mill wherein the solids content of the suspension is higher (more than 40 % by weight) that the shearing responsible for the particle size reduction takes place.

For the reasons provided above, Applicants respectfully submit that the suspensions of the instant invention are substantially different from the suspension disclosed in WO '330, and a rejection of claims 22-29 under 35 U.S.C. §102(b) should not be maintained. Likewise, Applicants respectfully submit that claims 22-29 are not properly rejected under 35 U.S.C. §103 for the following reasons.

WO '330 discloses slurries with a high solids content (40 to 60% by weight) having a viscosity less than 1000 cps. (1 Pa.s). There is no incentive in WO '330 for the skilled artisan to adapt the slurries disclosed therein in order to obtain slurries having a solids content of 10-40% by weight, having viscosities lower than 0.04 Pa.s, and exhibiting a quantity of silica in the supernatant that is more than 50% after centrifuging the suspension at 7500 rpm for 30 minutes, as is provided by the present invention.

Furthermore, WO '330 actually teaches away from slurries as disclosed in the present invention. For instance, on page 10, line 22, it is mentioned that slurries obtained after the step of high density milling require a mild agitation to prevent settling. Moreover, on page 10, line 26, it is mentioned that "some settling of the silica may still occur" (despite the agitation).

Because of the general tendency of silica slurries to settle, and the clear tendency of the slurries disclosed in WO '330 to settle, the skilled artisan would not have been motivated to follow the disclosure of WO '330 for the purpose of obtaining stable slurries like those of the instant invention, i.e., having a high stability upon storage and being in the form of a pumpable liquid. Indeed, the Federal Circuit has clearly established that a reference must be considered in [its] entirety and full consideration must be given to disclosures in the reference that diverge from and teach away from the invention at hand. W.L. Gore & Associates, Inc. v. Garlock, Inc., 220 USPQ 303, 311 (Fed. Cir. 1983).

Further, with regard to stability, Applicants also respectfully point out that the silica slurries of the present invention present a dramatic improvement over slurries of the prior art.

Besides teaching away, the above citations from WO '330 also indicate that the slurries of that disclosure do not have the properties of the slurries of the present invention, i.e., high stability upon storage. In contrast, the stability of the claimed slurries has been demonstrated by the settling test under centrifugation (see page 21, line 7, for example).

The level of stability demonstrated by the slurries of the present invention is, in fact, an unexpected improvement over slurries achieved in the prior art. Where there was, at the time the invention was made, no known or obvious method of making the claimed composition, or where the claimed composition is found to possess unexpected characteristics, a conclusion of obviousness based on similarity of structure and gross characteristics between the [products] is rebutted. In re Wakefield, 164 USPQ 636 (CCPA 1970) discussed and distinguished in Ex Parte Gray, 10 USPQ2d 1922, 1926 (PBAI 1989).

For these reasons, reconsideration and withdrawal of the rejection of claims 22-29 under 35 U.S.C. §102 or 35 U.S.C. §103 in view of WO '330 is respectfully requested.

Beginning on page 3 of the Office Action, claims 22-37 and 39-45 were rejected under 35 U.S.C. §103 as allegedly being unpatentable over Chevallier et al. '570. Specifically, the Office Action maintains that the aqueous silica suspensions and methods of the instant claims would allegedly be obvious in view of Chevallier, because Chevallier teaches a method of reacting silica and acid in the claimed concentrations with a deagglomeration step to make a silica solution. The Office Action also rationalizes that while the exemplified solution of Chevallier has a 4% concentration of silica, the reference suggests

that a concentration of 20% is desirable, and would be obviously pursued because such a concentrated solution "is easy to handle, ship and use." Applicants respectfully traverse.

Firstly, Applicants respectfully note that claim 39 has been amended by specifying in step C) that the deagglomerating step is conducted until a suspension of agglomerates having a median diameter  $D_{50}$  smaller than 5  $\mu\text{m}$  is obtained.

Chevallier teaches an aqueous suspension of precipitated silica as well as a method for its preparation. Namely, the method comprises the following steps:

- precipitating silica (steps (a), (b) and (c)) substantially in the same manner as in step A of the instant invention, so as to obtain a silica pulp;
- filtrating the pulp to provide a filter cake (col. 4, lines 22-23); and
- drying said cake (col. 4, lines 22-24, lines 29-34), the drying being performed preferably by spray-drying, wherein the cake to be dried has a dry solids content greater than 18% by weight.

Between the filtration step and the drying step, the filtration cake may be subjected to a disintegrating operation (col. 4, lines 57-59), for example by transferring the cake into a colloidal or ball-type mill (col. 4, lines 57-59).

The product recovered after the drying step is eventually ground (col. 4, lines 66-67), and a precipitated silica is obtained "*advantageously in the form of a powder preferably having a mean size of from 5 to 70 microns*" (col. 5, lines 1-4).

Thus, the method of the instant invention is performed in a substantially different way with respect to the prior art method, namely as far as step C is concerned. Accordingly, the

silica that is obtained as the end product is substantially different. Chevallier does not disclose a deagglomeration step which is conducted so as to obtain a suspension with a particle size as disclosed in the instant invention. At the very most, Chevallier teaches that at the end of steps A) to C), the precipitated silica in the form of powder has a median diameter between 5 and 70  $\mu\text{m}$ .

Applicants respectfully submit that the objective of Chevallier is not to provide a silica suspension having a low viscosity, but a precipitated silica in solid form that may be used as a reinforcing filler for elastomers.

Applicants also respectfully stress that the mean particle size as well as the particle size distribution obtained in the deagglomeration step of the instant invention is mainly responsible for the low viscosity of the silica suspension obtained as the end product of the instant invention. Moreover, Chevallier does not provide any incentive for the skilled person to conduct a deagglomeration stop in a way so as to obtain a silica suspension with a low viscosity as claimed in the instant invention.

Therefore, Applicants respectfully submit that the features of the silica suspension of the instant invention, namely its low viscosity as well as the sedimentation properties thereof, are in no way suggested by Chevallier. Reconsideration and withdrawal of the rejection of claims 22-37 and 39-45 under 35 U.S.C. §103 over Chevallier et al. is respectfully requested.

Finally, on page 4 of the Office Action, claims 38 and 46 have been rejected under 35 U.S.C. §103 as being unpatentable over Chevallier as applied to claims 22-37 and 39-45 above, and further in view of Cox et al. Specifically, the Office Action maintains that the

cited claims are allegedly unpatentable because it would be obvious to wash the product of Chevalier with the organic solvent of Cox because doing so makes the "pure material desired by Chevalier."

Again, Applicants respectfully submit that the material "desired by Chevalier" is quite different from the suspension of the present invention. Chevallier teaches a precipitated silica in the form of powder which has a median diameter between 5 and 70  $\mu\text{m}$ . In contrast, the present invention provides for an aqueous suspension wherein the median diameter of the silica particles is less than 5  $\mu\text{m}$ . There is no overlap between the median diameter of silica particles taught in Chevallier and that taught in the present invention. Furthermore, lower viscosity is an important consequence of the median diameter disclosed in the present invention. Cox does not make up for this basic deficiency. Accordingly, reconsideration and withdrawal of the rejection of claims 38 and 46 under 35 U.S.C. §103 over Chevalier in view of Cox is respectfully requested.

This Amendment is believed to be a complete response to the Office Action dated December 8, 1997. Accordingly, a Notice of Allowance appears to be next in order. Should

Application Serial No. 08/765,901  
Attorney's Docket No. 004900-148

the Examiner have any questions concerning this amendment or the subject application, a telephone call to the undersigned would be appreciated.

Respectfully submitted,

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Date: April 8, 1998